PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:	09/918,158
Filing Date:	July 30, 2001
Applicant:	Robert A. Dichiara Jr.
Group Art Unit:	1731
Examiner:	Christopher A. Fiorilla
Title:	OXIDE BASED CERAMIC MATRIX COMPOSITES
Attorney Docket:	7784-000146

Director of the United States Patent and Trademark Office
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.131

Sir:

I hereby declare under penalty of perjury as follows:

1. That I am the sole inventor of the above-identified application.

2. That the invention was conceived and at least partially reduced to practice in this country prior to December 15, 1999, the filing date of the United States Patent No. 6,497,776 to Butler et al.

3. That the invention was constructively reduced to practice, as vid方 by the invention disclosure document attached as Exhibit A, prior to December 15, 1999.

4. That the invention has never been abandoned, suppressed, or concealed.

5. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are being made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, and patent issuing thereon, or any patent to which this verified statement is directed.

Dated: 2/2/04

Robert A. DiChiara, Jr.

McDonnell Douglas

Proprietary Employee Disclosure Record (Drawing and Description Sheet)

Intellectual Property Dept. Docket No. R-8916

Provide the following information c ncerning the disclosed item and in the indicated sequence:

1. Discuss the problems which the item is designed to solve, referring to any prior devices of a similar nature with which you may be familiar.
2. State the advantages of the item over presently known devices, systems or processes.
3. Specifically describe the item and its operation. You may use and attach copies of sketches, prints, photographs, and illustrations, which should be signed, witnessed and dated. Use numbers and descriptive names in descriptions and drawings.
4. List all known and other possible uses for the item.
5. List the features of the item that are believed to be novel.

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Oxide Based Ceramic Matrix Composite

Why CMC?

CMC is an emerging material that can be used in high temperature structural environments for aerospace and industrial applications. Advanced structural ceramics are materials that have relatively high mechanical strength at high temperatures. These materials face a number of demanding environmental conditions such as high temperature, corrosive, and high acoustic environments.

The oxide based ceramic matrix composite (CMC) developed is a economic a low dielectric, thermally stable, structural ceramic system good to > 2300°F using sol gel technology. The matrix can be reinforced with a variety of fibers. The system was developed for oxide fibers such as Nextel 720, but is not limited to this fiber. The CMC's primary advantage over carbon-carbon and other high temperature composites is its low cost, near net-shape manufacturing process.

History

Abbound (1995), Coors (1993) and Greenhunt (1994) point out that prior to 1980 ceramics were considered monolithic materials (made of one material). The advantages of monolithic ceramics is that the ceramic properties such as high strength, wear resistance, hardness, stiffness, corrosion resistance, thermal expansion and density can be varied depending on the starting materials. Deckman (1993) discussed that the density of ceramics are significantly lower (0.08 - 0.14 lb/in^3) compared to metallic counterparts (generally > 0.3 lb/in^3). Abraham (1990) discussed the problem with monolithic ceramics was their brittleness. If damaged, the monolithic ceramics had no ductility like metal, and instead would shatter (catastrophically fail). Catastrophic failure kept designers from considering advanced structural ceramics in many structural applications.

Each sheet must be signed, dated and witnessed. Signature(s) f employee(s/inventor(s) (use black ink)	Date	Witnessed, read and underst od by	Date
1. Robert A. DiChiara Jr.	[redacted]	Robert French	[redacted]
2.			
3.			

MDC 136-2 (REV 21 JAN 90)

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Abraham (1990), Ceramic Industry magazine (1990), and Eckert et. al (1990) discussed how in the mid-1980s a revolution in the field of ceramics took place with the development of new ceramic fibers (from Nippon Carbon and 3M) and the development of the French-SEP (Lasday, 1993) Chemical Vapor Infiltration process (CVI). Fibers were added to the ceramic matrix producing a fiber-reinforced ceramics which increased the ceramics strength, and toughness and eliminated (reduced) catastrophic failure at high temperatures. Each unique type of fiber added to the ceramic mix gave unique properties to the material. The exploration of fiber types and resulting properties leads to a number of combinations. Fiber mixes can be uniquely tailored to specific applications. These ceramics were known as ceramic matrix composite (CMC) or continuous-fiber-reinforced ceramic composites (CFCC) to help distinguish them from chopped fiber reinforced ceramics. The problem with the CVI process is it is expensive and slow to produce parts (processing time in months). The process is labor intensive, capital intensive, and limited with respect to the size and shape of parts.

In the 1990s a number of CMC organic-metallic processes were developed. These processes followed the same standard processing procedures and equipment developed for making organic composites, thereby eliminated many of the slow and costly limitations that were found with the CVI process. In this process ceramic fibers are first made and woven into cloths (like fiber glass or carbon fibers for organic composites). The flexible ceramic cloth is infiltrated with an organic-metallic matrix (like the epoxy matrix for organic composites), this impregnated cloth is placed on a complex tool, and processed under low pressure and low temperature (the process is known as autoclave). After autoclaving, a complex shaped ceramic structure is formed and then it is further heated in a furnace to finish the process. This process is most likely the way parts will be made in the future, but the product produced is inconsistent and the process will need further development.

The key to the strength and toughness of CMC system is to maintain limited amount of bonding of the fiber and the matrix. This is difficult to achieve considering the amount of thermal energy that is being applied to the surface chemistry to the matrix and the fiber surface. Success has been shown in four basic types of ceramic matrix system: (1) Chemical vapor infiltration (CVI), (2) glass ceramics, (3) organo-metallic derived from polymer precursors, and (4) oxide matrix ceramics.

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Glass ceramic CMC were one of the first systems developed. They start with glass powder, often formulated with silicates, which are thermoplastically formed along with reinforcing fabric at very high temperature and pressures. The fibers must be protected with fiber interface coatings in order to control fiber-matrix interface. The CMC is subjected to a free standing post cure to crystallize the matrix. Fiber interface coatings are susceptible to oxidation well below 1800°F. However, in a high densified system such as this, the fiber coatings are protected from the oxidizing environment. High strengths have been achieved with flat panels, however producability has greatly restricted its applications in three dimensional structures. Organic-metallic derived from polymer precursors are analogous to carbon-carbon. A polymer composite is fabricated, and then pyrolized to a ceramic. The volume loss during pyrolysis must be reinfiltrated with resin and pyrolized again. This process may be repeated up to ten times in order to achieve the densification necessary to provide oxidation protection to fiber coatings. The most common organic-metallic system used are Polysiazane, and Blackglas (Allied Signal). SiC fibers such as Nicalon are most commonly used with this system, along with fiber coatings such as boron nitride (BN). The disadvantage to this system are the cost, high dielectric constant and the susceptibility to the BN oxidation.

General Electric (GE) has become very strong in the CMC oxide matrix development with their system using binders, powers and silicone resins.

At McDonnell Douglas aluminum phosphate bonded alumina oxide CMC was developed in the mid-1980's. Fiber reinforcement was primary Nicalon 8 harness satin fabric. After 3 years of testing in November 1993 a CMC auxiliary power unit (APU) duct was flown on an F/A-18. This marked the first time that ceramic matrix composite has flown on a Navy aircraft. Studies of the matrix found repetitive cycles in excess of 1500°F causing phase inversions in the matrix and limited the material use to 1400°F. Due to the inherent thermally instability in the aluminum phosphate system a sol-gel oxide matrix system with new oxide fibers by 3M were developed to overcome the temperature and strength limitations of the aluminum phosphate system yet still allow the low cost processing methods developed for organic composites.

STOP

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1. Robert A. DiChiara Jr.	[redacted]	Robert Farrell	[redacted]
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